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Three issues have been addressed in this report. Firstly, the biaxial shear dominated failure properties of graphite/polymid fabric composites have been investigated at room temperature using the biaxial Iosipescu method. Secondly, a new testing procedure has been suggested for evaluating micro-damage in polymer matrix composites. Using this method, three-dimensional failure characteristics of damage generated in the graphite/polyimid Iosipescu specimens can be determined for various loading conditions. Finally, non-linear finite element computations of internal stresses in the Iosipescu specimens have been performed taking into account the effect of specimen sliding within the Iosipescu fixture.

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BIAXIAL FAILURE ANALYSIS OF GRAPHITE REINFORCED POLYIMIDE COMPOSITES*

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Introduction

High temperature polymers and polymer matrix composites (PMC's) are finding increasing use in the aerospace and electronics industries. Graphite fiber reinforced polyimides are advanced thermosetting polymer composites which have become highly relevant in the realm of high temperature applications (ref. 1). These composites possess exceptional specific properties and are stable to temperatures as high as 360 °C (680 °F). For successful application of graphite/polyimide composites, it is essential to have a reliable database of material properties and a detailed understanding of fracture and failure behavior, especially at elevated temperatures. A major limitation of many fiber/polymer composite systems is the inability of these materials to resist intralaminar and interlaminar damage initiation and propagation under biaxial, shear dominated, monotonic and cyclic loading conditions. The purpose of this research is to investigate failure properties of fabric and unidirectional graphite/polyimide composites tested over the temperature range -50 °C to 315.6 °C (-58 °F to 600 °F) under biaxial, shear dominated loading conditions.

The Iosipescu shear test, which was originally designed for determining shear properties of metals (ref. 2), was first applied to composite materials by Walrath and Adams (ref. 3). The traditional Iosipescu shear test essentially consists of a double edge-notched beam specimen, to which two counteracting force couples are applied such that the net bending moment at the specimen midlength is zero, and a relatively uniform shear stress field exists in the central gage section of the specimen. Based upon the traditional Iosipescu shear test, an in-plane biaxial Iosipescu test fixture has been designed and developed at the University of Cambridge (ref. 4). The fixture (Fig. 2) is capable of testing Iosipescu specimens in either pure shear or a combination of shear and transverse tension/compression under static or cyclic loads. Shear tests can be performed under externally applied compressive loads (P_0) normal to the longitudinal axis of the specimen as in the traditional test (refs. 1 and 2). For in-plane, biaxial tests, the specimen is rotated clockwise (c.w.) or counter-clockwise (c.c.w.) such that the compressive load (P_α) is applied at various angles (α) to the normal (Fig. 3).

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Recent studies have employed the fixture for obtaining biaxial failure and fracture properties of unidirectional GRP and CFRP composites, teak wood, Ti/SiC composites and adhesive joints (refs. 4-8). For this study, a series of shear and biaxial Iosipescu tests were performed at room and elevated temperatures on the following three composite materials:

1. Graphite/PMR-15 (fabricated at the NASA Lewis Research Center); Fiber: T650-35, 8-H Satin Cloth; Ply Layup: 16-ply (warp-aligned); Matrix: PMR-15.
2. Graphite/Avamid-R (fabricated by DuPont); Fiber: T650-35, 8-H Satin Cloth; Ply Layup: 10-ply (warp-aligned and 0/90); Matrix: Avamid-R.
3. Graphite/PMR-15 (fabricated at the NASA Lewis Research Center); Fiber: T650-35, unidirectional; Ply Layup: 34-ply; Matrix: PMR-15.

Composites 1 and 3 were fabricated using a simulated autoclave according to G.E.'s specifications. The panels were post-cured according to G.E.'s schedule. In this paper, only the results from the biaxial testing of the fabric composites are presented.

The load-displacement diagrams for the graphite/PMR-15 Iosipescu specimens tested in the biaxial fixture at room temperature are presented in Fig. 4. It can be observed that the shape of the load-displacement curves depends on the loading angle. Similar tests were performed on the graphite/Avamid-R composites (warp-aligned and 0/90). The loads at failure as a function of the loading angle (α) for the investigated composites are shown in Fig. 5. At room temperature, the biaxial strength properties of these three composite systems appear to be different. Moreover, different failure modes were identified and it was also established that the shear and biaxial strength properties of the graphite/PMR-15 system may be significantly influenced by the manufacturing process.

The micro-failure process in the PMR-15 system tested at room temperature starts from the initiation of interlaminar cracks between the layers (Fig. 6a). This usually leads to the formation of large delaminations within the gage section of the Iosipescu specimens. Often, one of the interlaminar cracks propagates catastrophically along the sample causing a significant drop on the load-displacement curves. Such load drops can be observed in Fig. 4. This effect, however, seems to be dependent on the manufacturing process. If the strength of the interfaces between the layers is high, the load drops do not occur. The interlaminar damage in the PMR-15 system creates large out-of-plane deformations on the specimen surface i.e., bulging (see Fig. 6b). The micro-failure process which determines the failure of the Avamid-R system is the formation of intralaminar cracks along the notch root axis. The second mechanism is the formation of interlaminar cracks between the layers. Since the interlaminar strength properties of the Avamid-R system appear to be higher compared to the PMR-15 system, the bulging effect on the composite surface is substantially less pronounced.

The micro-damage generated in the composite Iosipescu specimens tested under biaxial loading conditions can be evaluated by capturing and performing qualitative analyses of scanning electron microscope (SEM) images from planar specimen slices. Subsequently, the slices are reassembled into 3-D space and the net volumetric effect of damage can be determined. In Fig. 7, a 3-D projection of damage within the gage section of the PMR-15 Iosipescu specimen tested in shear is presented.

The biaxial tests at elevated temperatures were performed using a high temperature setup assembled at the University of Denver (Fig. 8). Since the high temperature, biaxial Iosipescu research is still in progress, only preliminary results from the high temperature investigation are presented and discussed in this paper. As an example, the load-displacement diagrams for the graphite/PMR-15 and graphite/Avamid-R (0/90) composites tested in shear at various temperatures are shown in Figs. 9 and 10, respectively. The effect of elevated temperature on the load-displacement curves for the graphite/Avamid-R composite is significantly stronger in comparison with the graphite/PMR-15 system. From the maximum loads determined using the curves in Figs. 9 and 10, the shear strength properties were subsequently estimated. The shear strengths of the composites as a function of temperature are presented in Fig. 11. At room temperature, it appears that the shear strength of the graphite/Avamid-R system is slightly higher than that of the graphite/PMR-15 system. However, at elevated temperatures above 232 °C (450 °F), the graphite/Avamid-R composite exhibits a rapid decrease in its shear strength, whereas the shear strength of the PMR-15 system gradually decreases with an increase in temperature.

References

1. Meador, M.M.; Cavano, P.J.; and Malarik, D.C.: High Temperature Polymer Matrix Composites for Extreme Environments, Structural Composite: Design and Processing Technologies. Proc. Sixth Annual ASM/ESD Advanced Composites Conference, Detroit, Michigan, pp. 8-11, October 1990.

2. Iosipescu, N.: New Accurate Procedure for Single Shear Testing. J. Materials, vol. 2, no. 3, 1967, pp. 537-566.

3. Walrath, D.E.; and Adams, D.F.: The Iosipescu Shear Test as Applied to Composite Materials. Exp. Mech., vol. 23, no. 1, 1983, pp. 105-110.

4. Broughton, W.R.; Kumosa, M.; and Hull, D.: Analysis of the Iosipescu Shear Test as Applied to Unidirectional Carbon-Fiber Reinforced Composites. Comp. Sci. Tech., vol. 38, 1990, pp. 299-325.

5. Bansal, A.; and Kumosa, M.: Experimental and Analytical Studies of Failure Modes in Iosipescu Specimens Under Biaxial Loadings. J. Comp. Mat., vol. 29, no. 3, 1995, pp. 334-358.

6. Bansal, A.; and Kumosa, M.: Application of the Biaxial Iosipescu Method to Mixed-Mode Fracture of Unidirectional Composites. Int. J. Fracture, vol. 71, 1995, pp. 131-150.

7. Ding, S.: Mixed Mode Failure Analysis of Adhesively Bonded Composite Systems Using the Modified Iosipescu Test Method, Ph.D. Thesis, Oregon Graduate Institute of Science and Technology, Portland, Oregon, April 1996.

8. Balakrishnan, M.V.; Bansal, A.; and Kumosa, M.: Biaxial Testing of Unidirectional Carbon-Epoxy Using the Biaxial Iosipescu Test Fixture. J. Comp. Mat., vol. 31, no. 5, 1997, pp. 486-508.

Objectives

- Determine failure and fracture properties of unidirectional and fabric graphite reinforced polyimide composites based on PMR-15 and Avamid-R resins tested under biaxial, shear dominated loading conditions over the temperature range -50 °C to 315.6 °C (-58 °F to 600 °F)
- Characterize micro-damage initiation and development in the composites as a function of:
  - Testing temperature
  - Biaxial, shear dominated in-plane stress conditions
  - Type of polyimide resin
  - Manufacturing process
- Develop a finite element model of biaxial, graphite/polyimide Iosipescu specimens for elevated temperature applications

Fig. 1

Biaxial Iosipescu Test Fixture

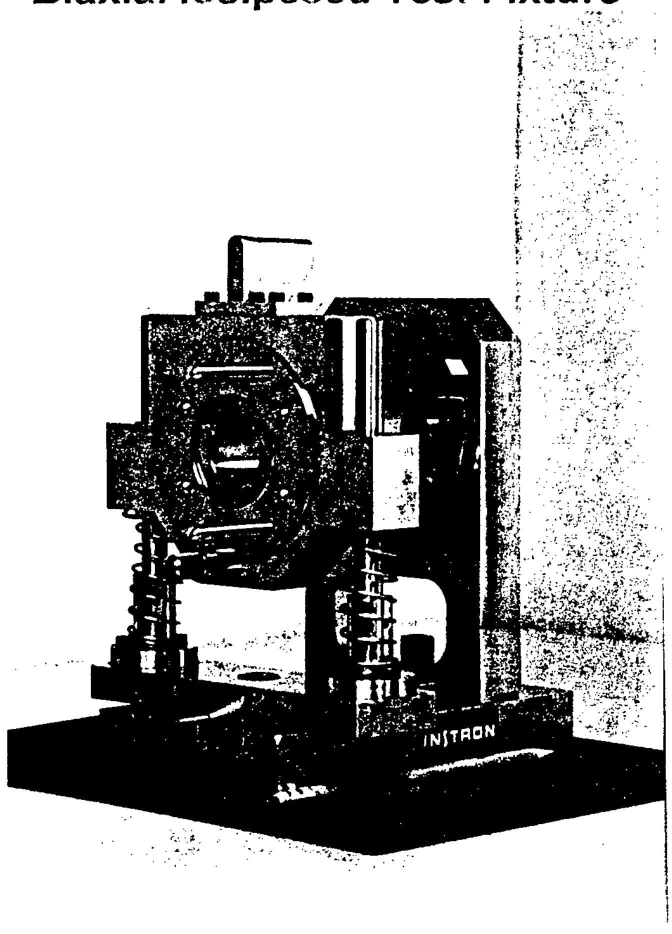

Fig. 2

Loading Diagram for Inducing an In-Plane Biaxial Stress State

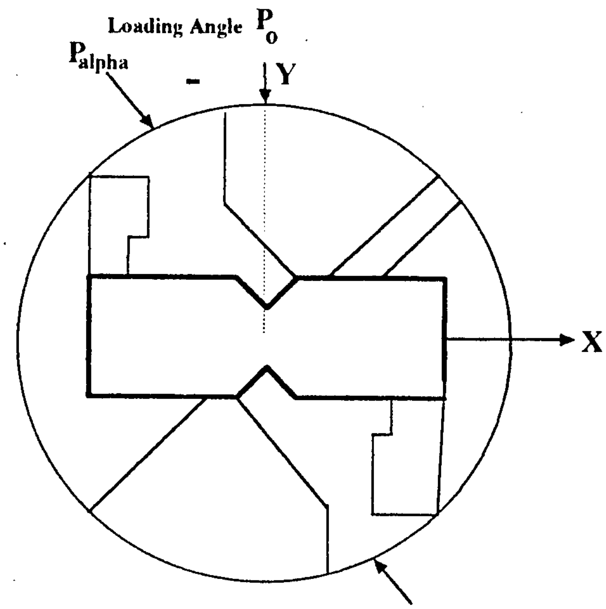

Fig. 3

Load as a Function of Displacement for Graphite/PMR-15 Under Biaxial Conditions

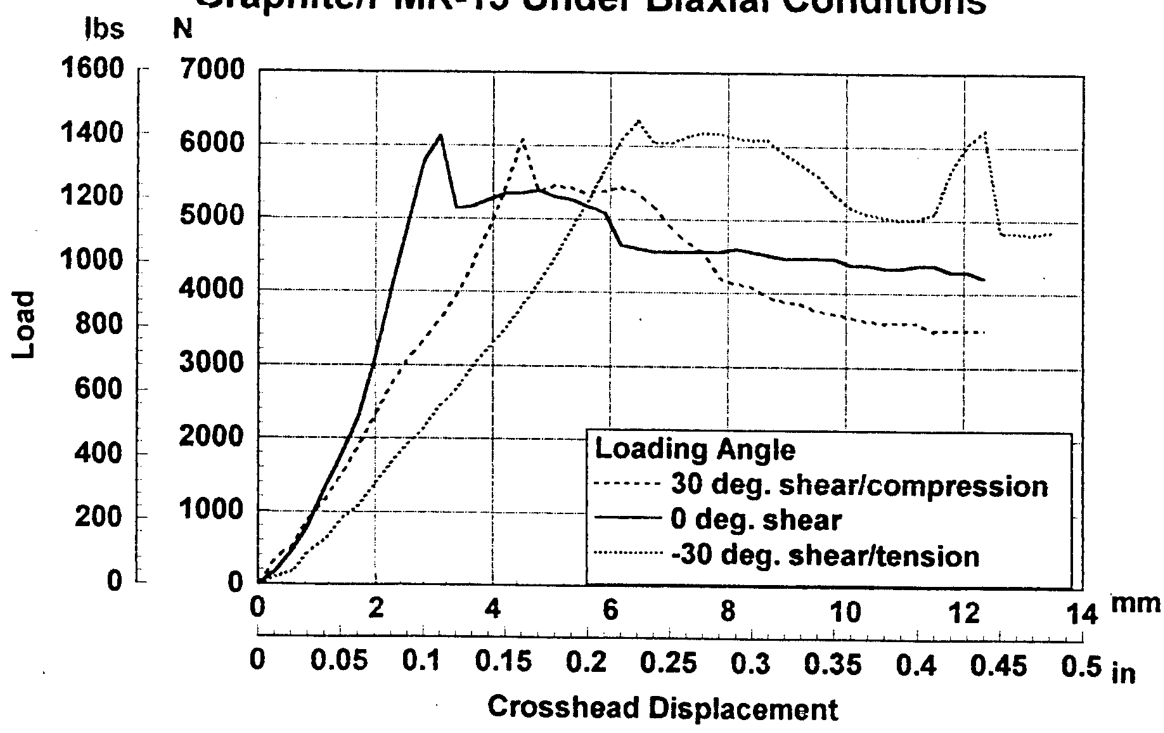

Fig. 4

Loads at Failure as a Function of Loading Angle for Graphite/PMR-15 and Graphite/Avamid-R

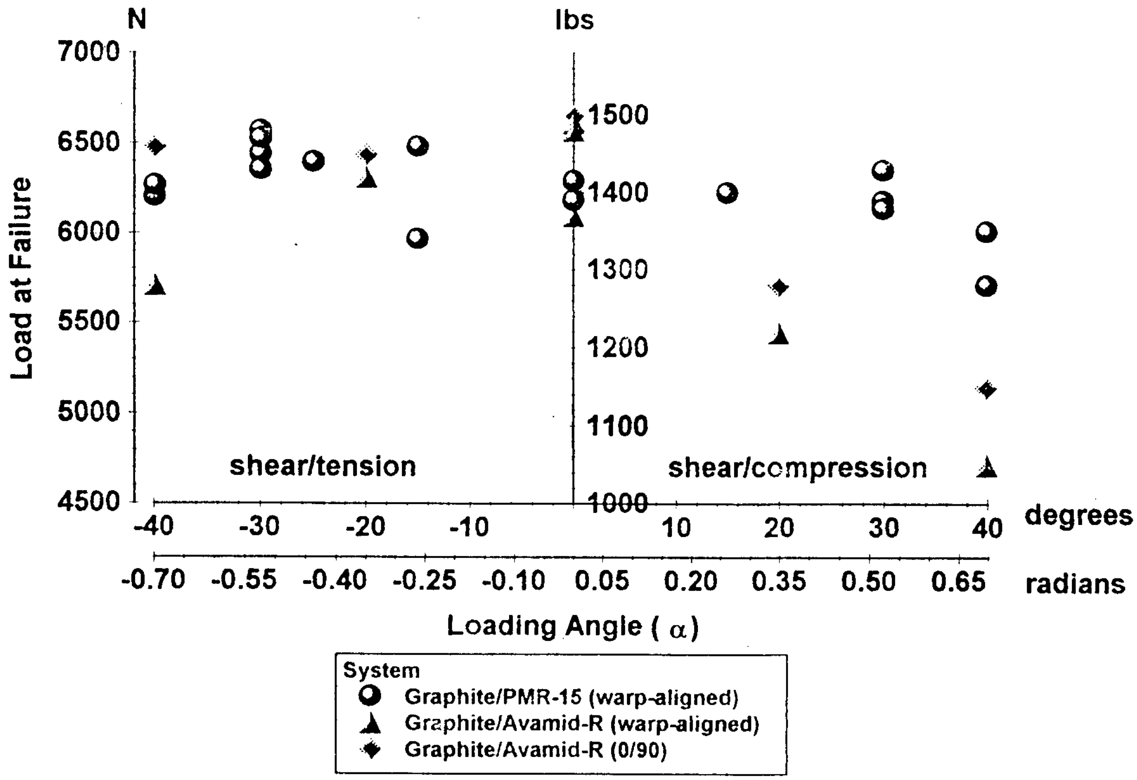

Fig. 5

Damage at the Notch Root in Graphite/PMR-15 Iosipescu Specimens: a) Inter- and Intralaminar Cracks

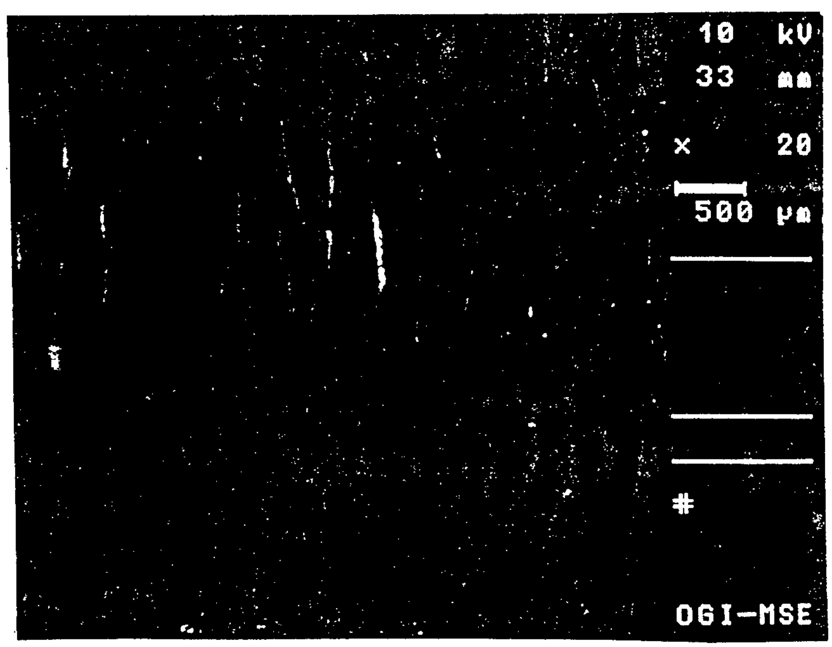

Fig. 6a

Damage at the Notch Root in Graphite/PMR-15 Iosipescu Specimens: b) Out-of-Plane Damage (Bulging)

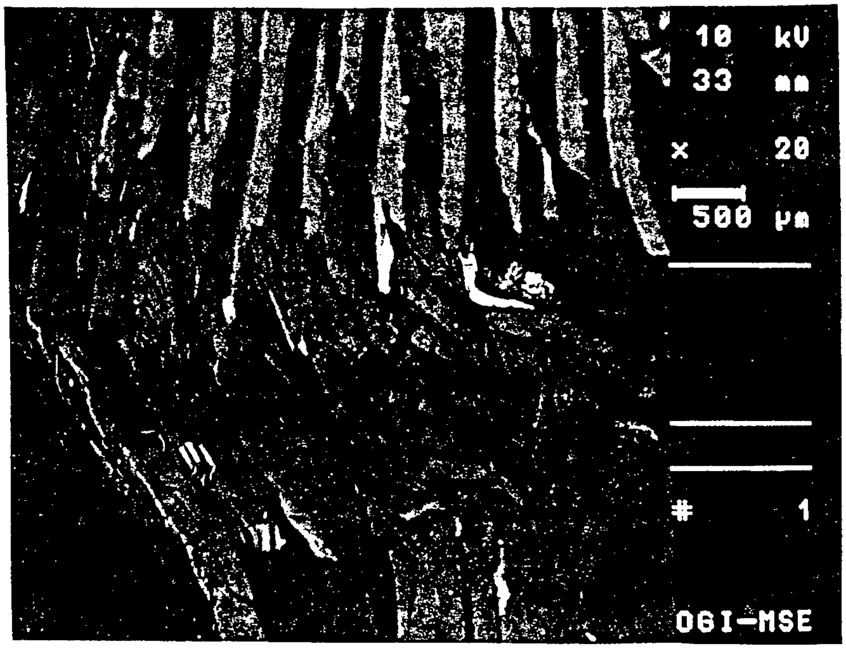

Fig. 6b

3-D Projection of Damage in a Graphite/PMR-15 Iosipescu Specimen Tested in Shear at Room Temperature

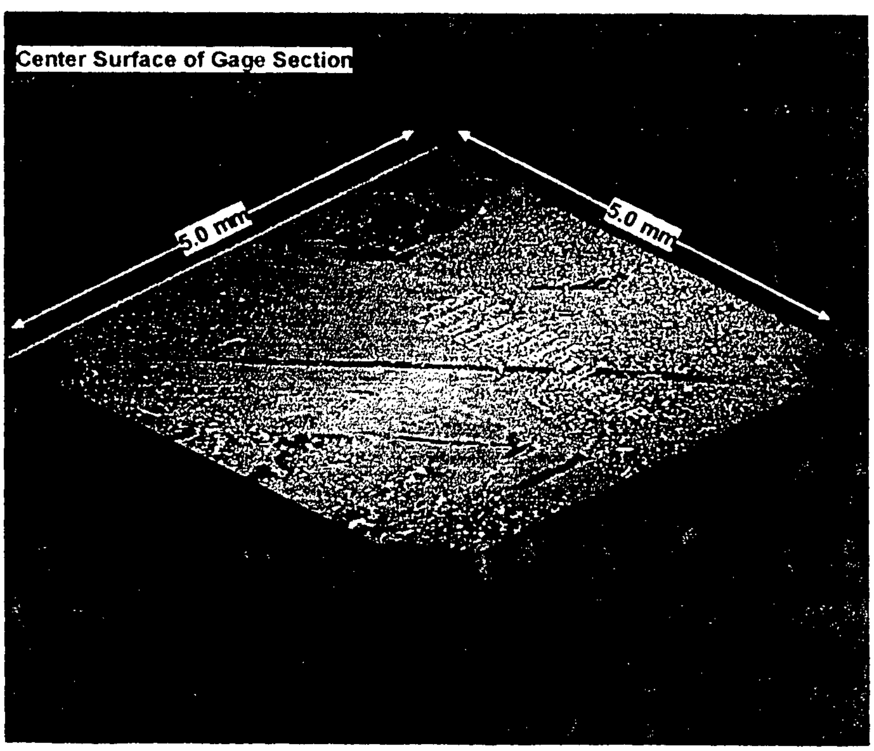

Fig. 7

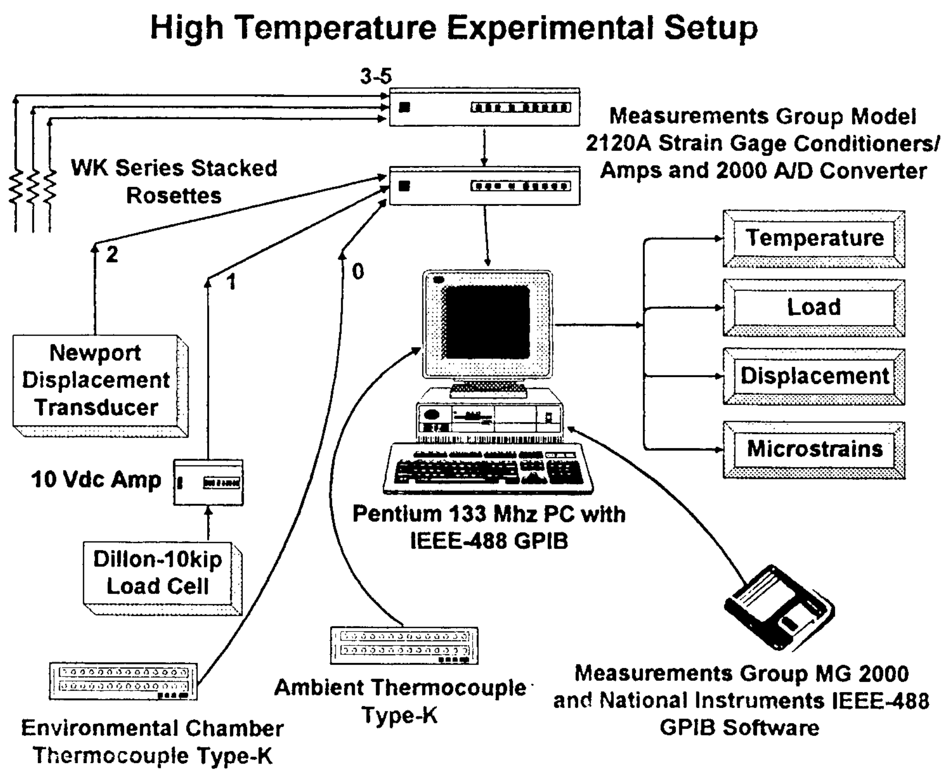
High Temperature Experimental Setup
3-5
Measurements Group Model 2120A Strain Gage Conditioners/ Amps and 2000 A/D Converter
WK Series Stacked Rosettes
2
1
0
Temperature
Load
Displacement
Microstrains
Newport Displacement Transducer
10 Vdc Amp
Pentium 133 Mhz PC with IEEE-488 GPIB
Dillon-10kip Load Cell
Ambient Thermocouple Type-K
Measurements Group MG 2000 and National Instruments IEEE-488 GPIB Software
Environmental Chamber Thermocouple Type-K

Fig. 8

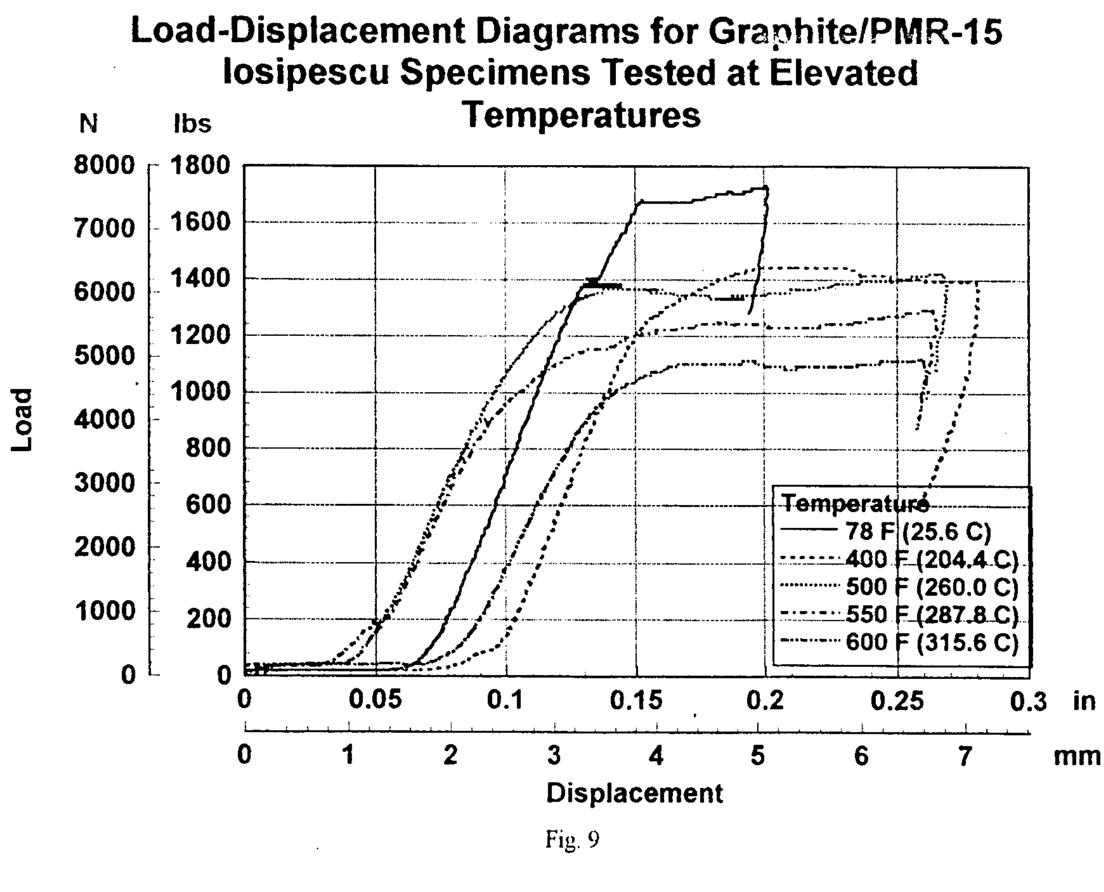
Load-Displacement Diagrams for Graphite/PMR-15 Iosipescu Specimens Tested at Elevated Temperatures
N
lbs
8000
7000
6000
5000
4000
3000
2000
1000
0
1800
1600
1400
1200
1000
800
600
400
200
0
Load
Temperature
78 F (25.6 C)
400 F (204.4 C)
500 F (260.0 C)
550 F (287.8 C)
600 F (315.6 C)
0
0.05
0.1
0.15
0.2
0.25
0.3
in
0
1
2
3
4
5
6
7
mm
Displacement

Fig. 9

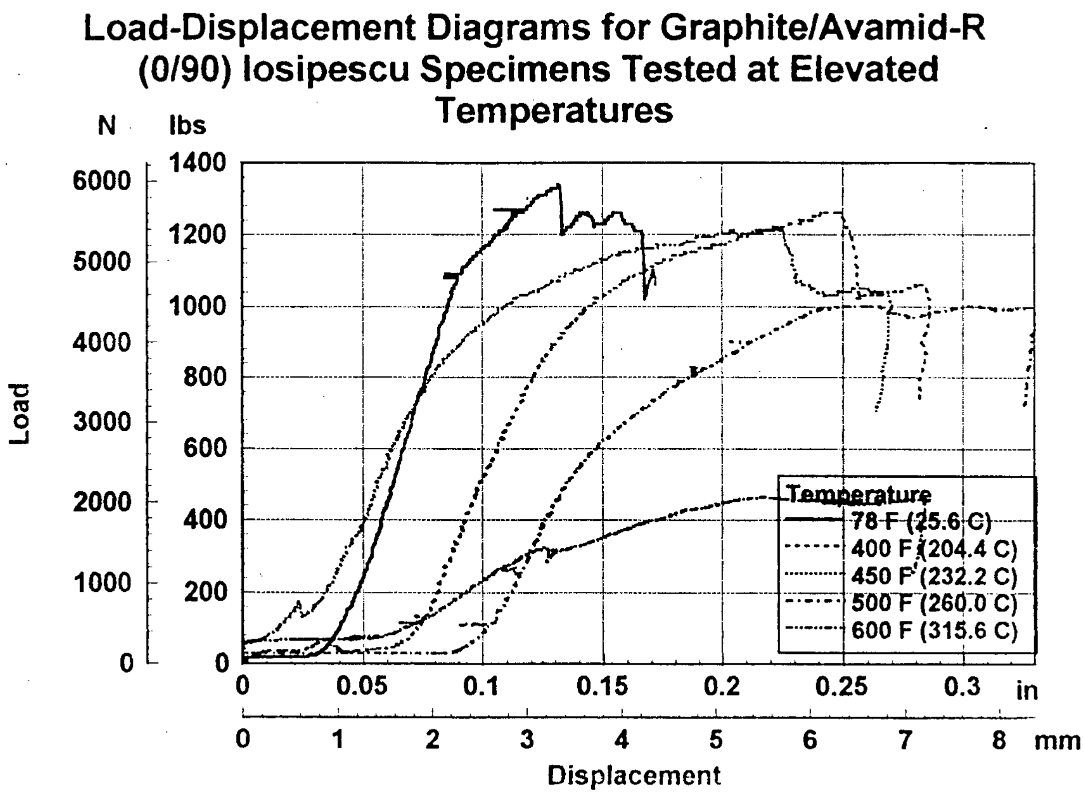
Load-Displacement Diagrams for Graphite/Avamid-R (0/90) Iosipescu Specimens Tested at Elevated Temperatures
N
lbs
Load
Displacement
in
mm
Temperature
78 F (25.6 C)
400 F (204.4 C)
450 F (232.2 C)
500 F (260.0 C)
600 F (315.6 C)

Fig. 10

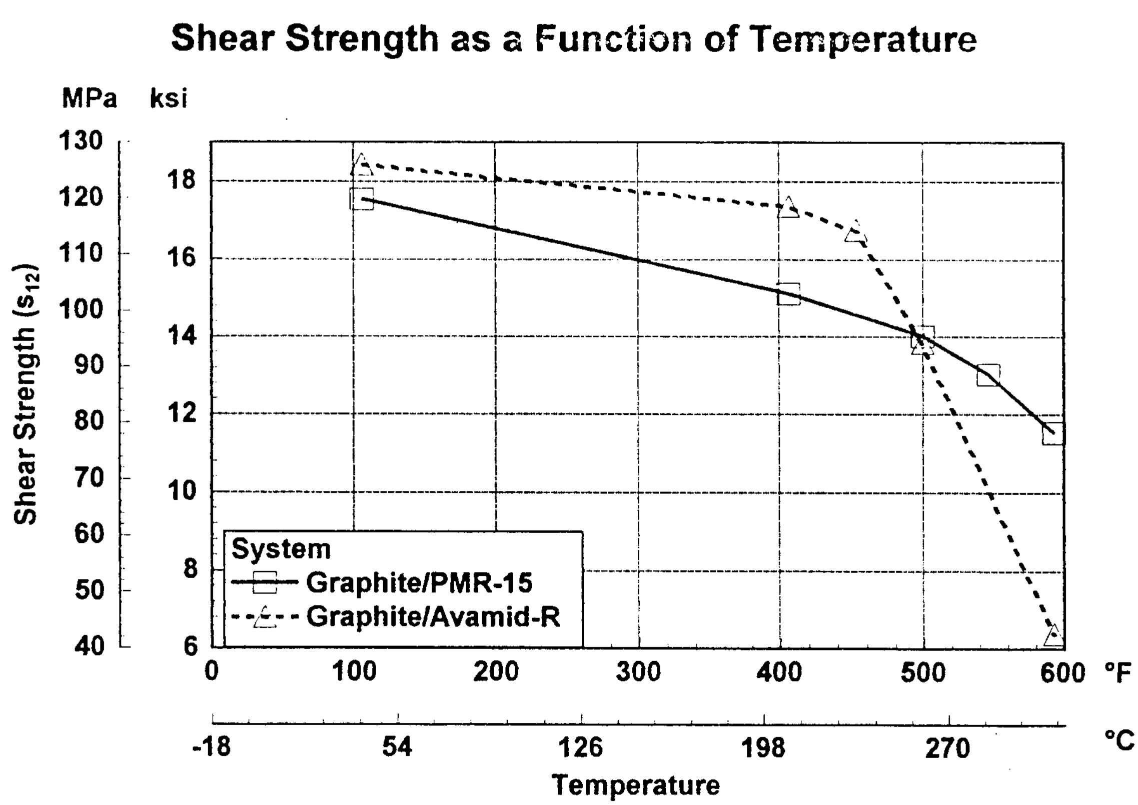
Shear Strength as a Function of Temperature
MPa
ksi
Shear Strength (s_{12})
Temperature
°F
°C
System
Graphite/PMR-15
Graphite/Avamid-R

Fig. 11

Summary of Results

- Biaxial strength properties and failure modes for graphite/PMR-15 and graphite/Avamid-R are different at room temperature:
  - It appears that the shear strength properties of the Avamid-R system are better than the PMR-15 system
  - Under biaxial shear/compression loading conditions, the PMR-15 composite exhibits significantly higher strength in comparison with the Avamid-R system
- The effect of elevated temperatures on the load-displacement curves in shear for the graphite/Avamid-R composite is significantly more prevalent than for the graphite/PMR-15 composite
- The shear strengths of the Avamid-R system at elevated temperatures are significantly lower than the high temperature shear strengths of the graphite/PMR-15 system

Fig. 12

Conclusion

- Application of the biaxial Iosipescu test fixture can be successfully extended to include graphite reinforced polyimide fabric and unidirectional composites when determining:
  - shear strength properties at room and elevated temperatures
  - shear dominated, biaxial failure mechanisms

Fig. 13

Future Work

- Complete biaxial Iosipescu testing on fabric and unidirectional graphite polyimide composites over a range of temperatures
- Complete development of image analysis techniques for reproduction of damage from planar specimen slices
- Develop acoustic emission and resonant frequency methodologies for monitoring and identifying damage progression in the composites at high temperatures
- Develop numerical schemes for modeling damage in composite biaxial Iosipescu specimens at high temperatures

Fig. 14